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## Work with the Mass Spectrometer

The empty graphite crucible was run to determine the background for the 27, 28, and 29 peak. The data are shown in Table I. Again, above 1600°K the temperature control was difficult. The background for each peak increases monotonously as the temperature is increased. The ratio of 28 to 29 peak is again 70.

To avoid temperature fluctuations at high temperature, the empty tungsten crucible was run; the data are shown in Table II. The background is somewhat more intense than with the graphite crucible, due probably to the fact that the spectrometer was not degassed sufficiently. The background increases the same way with temperature as when the graphite crucible was used. The ratio of 28 to 29 peak is again around 70 indicating that the graphite crucible did not contribute, noticeably, any organic material with mass 29 to the background. The temperature control, however, was much better.

Finally, aluminum nitride was introduced into the tungsten crucible and the run made again with increasing temperature. The data are shown in Table III. As expected, at the higher temperatures the peak heights do increase, due to the vaporization of aluminum nitride. If the intensity of both peaks 28 and 29 is corrected for the background, even in run 4 the ratio remains 70. This error (70 instead of 138) must be due to the instrument, because only nitrogen and aluminum come out from the Knudsen cell.

The reason for the low 28 to 29 ratio can be due to the electrometer. A change of two scales in the milimicroamps range is carried out, while changing from the 28 to 29 peak. If each scale has an error of 25%, the ratio could drop down to 88 from 138. Furthermore, as the peak with low intensity (29) is to the high mass side from the high intensity peak (28), the background due to 28 is not corrected sufficiently. The peaks have shapes of a velocity distribution, with a fairly long tail toward high mass numbers.

It would be possible to use this experimental ratio of 70 as a calibration. This would, however, require calibration at higher $N^{15}$ to $N^{14}$ ratios, that is, prepare mixtures, and run a calibration curve. As this is tedious and less accurate, a great effort will be spent in the near future to obtain the correct ratio by checking the electrometer and the background.

Further inspection of Table III shows that the aluminum coming from the Knudsen cell condenses on the shutter when the latter is closed, but the nitrogen does not. Run 2, where the temperature is low enough so that only background shows, gives the same 27, 28, and 29 intensity, shutter open or closed. Run 4, where the temperature is high enough to cause vaporization of AlN, the 27 peak drops in intensity to the background when the shutter is closed. The intensity of peak 28 and 29 hardly changes when the shutter is closed; the nitrogen does not condense on the shutter but passes around it into the source. There is a minute drop in intensity of peaks 28 and 29 when the shutter is closed; by passing around the shutter, not the same number of nitrogen atoms reach the source from the Knudsen cell as when there is nothing in the path of the gas. It is surprising how inefficient the shutter is from keeping gas from the Knudsen cell from reaching the source.

In the future, besides trying to find the correct $N^{14}$ to $N^{15}$ ratio, the preparation of $AlN^{15}$ will be started.

## Iridium - Carbon System

One preliminary run was carried out with a 10 at% C sample at 1500°C. The X-ray diffraction pattern of iridium did not show any noticeable change compared to that of pure iridium. Higher temperatures are probably required for reaction to take place.

At the present, the modifications are made in our high temperature camera, to reach 2200°C.

TABLE I

Determination of Background of

Empty Graphite Crucible, 27, 28, and 29 Peaks

| Temp. °K | log $A_{27}$ | log $A_{28}$ | log $A_{29}$ |
|---|---|---|---|
| 300 | 2.095 | 4.555 | 2.702 |
| 1293 | 2.272 | 4.576 | 2.725 |
| 1508 | 1.88 | 4.605 | 2.864 |
| 1593 | 2.360 | 4.646 | 2.894 |
| 1858 | 3.057 | 5.691 | 3.987 |
| 1849 | 2.736 | 5.359 | 3.521 |
| 1708 | 2.496 | 4.825 | 3.070 |
| 1638 | 2.375 | 4.747 | 2.994 |
| 1748 | 2.435 | 4.900 | 3.150 |
| 1538 | 2.300 | 4.675 | 2.927 |
| 1623 | 2.295 | 4.743 | 2.938 |
| 1678 | 2.360 | 4.800 | 3.058 |
| 1273 | 2.017 | 4.518 | 2.742 |
| 300 | 2.082 | 4.775 | 2.796 |

TABLE II

Determination of Background of

Empty Tungsten Crucible, 27, 28, and 29 Peaks

| Temp. °K | log $A_{27}$ | log $A_{28}$ | log $A_{29}$ |
|---|---|---|---|
| 300 | 2.78 | 5.48 | 3.65 |
| 1293 | 3.04 | 5.46 | 3.69 |
| 1553 | 2.99 | 5.48 | 3.69 |
| 1733 | 3.09 | 5.54 | 3.70 |
| 1413 | 2.87 | 5.45 | 3.68 |
| 1888 | 3.22 | 5.58 | 3.79 |
| 1628 | 2.96 | 5.46 | 3.69 |
| 1833 | 3.04 | 5.49 | 3.75 |
| 1588 | 2.92 | 5.46 | 3.71 |
| 1688 | 2.92 | 5.45 | 3.68 |
| 1768 | 2.96 | 5.49 | 3.69 |
| 1858 | 3.04 | 5.52 | 3.74 |

TABLE III

Vaporization of Aluminum Nitride in Tungsten Crucible

| Run | Temp. °K | Area 27 Peak | 28 Peak | 29 Peak | log $A_{27}$ | log $A_{28}$ | log $A_{29}$ |
|---|---|---|---|---|---|---|---|
| | 300 | | | | 2.676 | 5.447 | 3.61 |
| 1 | 1453 | 1240 | 334000 | 5250 | 3.094 | 5.524 | 3.72 |
| 2 | 1580 | 1900 | 374000 | 5690 | 3.280 | 5.573 | 3.76 |
| Shutter closed | 1580 | 1930 | 368000 | 5010 | 3.285 | 5.566 | 3.70 |
| 3 | 1672 | 3770 | 387000 | 5080 | 3.576 | 5.588 | 3.71 |
| 4 | 1810 | 32900 | 912000 | 12200 | 4.515 | 5.960 | 4.09 |
| shutter closed | 1810 | 1590 | 834000 | 10900 | 3.20 | 5.921 | 4.04 |
| 5 | 1567 | 1020 | 288000 | 3540 | 3.01 | 5.460 | 3.55 |
| 6 | 1230 | 734 | 277000 | 3950 | 2.865 | 5.444 | 3.60 |
| 7 | 1282 | 637 | 264000 | 3720 | 2.805 | 5.420 | 3.57 |
| 8 | 1368 | 701 | 261000 | 4010 | 2.845 | 5.417 | 3.60 |
| 9 | 1535 | 973 | 275000 | 4030 | 2.990 | 5.440 | 3.61 |
| 10 | 1637 | 1870 | 296000 | 4070 | 3.272 | 5.472 | 3.61 |
| 11 | 1733 | 5950 | 408000 | 3320 | 3.775 | 5.61 | 3.52 |
| 12 | 1690 | 3690 | 322000 | 3590 | 3.565 | 5.508 | 3.55 |

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